REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
		FINAL REPORT 01 Apr 95 - 31 Mar 96

4. TITLE AND SUBTITLE
Growth and Structure of MBE-Deposited Iridium Silicide

5. FUNDING NUMBERS
61102F
2305/FS

6. AUTHOR(S)
Professor Falco

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Arizona Research Laboratories
University of Arizona
Tucson, AZ 85721

AFOSR-TR-96
0561

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)
AFOSR/NE
110 Duncan Avenue Suite B115
Bolling AFB DC 20332-0001

10. SPONSORING / MONITORING AGENCY REPORT NUMBER
F49620-95-1-0260

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION / AVAILABILITY STATEMENT
APPROVED FOR PUBLIC RELEASE: DISTRIBUTION UNLIMITED

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

We were able to form pure TrSi3 films at temperatures as low as 450oC, which is almost 200oC lower than previously reported. With our MBE growth techniques we also found a previously-unreported c-axis epitaxial TrSi3 growth mode at 700oC, found that the IrSi3 epitaxy on Si (111) was dominated by a Mode B orientation which had not previously been reported in the literature, as well as showed that the epitaxial growth of IrSe3 on Si(111) was superior to that on Si(100).

14. SUBJECT TERMS			15. NUMBER OF PAGES
			16. PRICE CODE
17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	

Grant No. AFOSR-F49620-95-1-0260
Growth and Structure of MBE-Deposited Iridium Silicide
Technical Contact: Lt. Col. Gernot S. Pomrenke

Charles M. Falco, P.I.
Arizona Research Laboratories
University of Arizona
Tucson, AZ 85721

Final Technical Report for Period 1 April 1995–31 March 1996

September 9, 1996

ABSTRACT

This report summarizes accomplishments of a four-year a research program, with emphasis on the final one-year period 1 April 1995–31 March 1996, to prepare iridium silicide films by Molecular Beam Epitaxy—MBE, and characterize their physical and chemical structure in detail as a function of preparation conditions using the wide variety of probes available in our laboratory. Many of these results were discussed in our previous Final Technical Report, dated 10 January 1995, as well as described in detail in various publications, so they are only summarized here. We were able to form pure $IrSi_3$ films at temperatures as low as 450 °C, which is almost 200 °C lower than previously reported. With our MBE growth techniques we also found a previously-unreported c-axis epitaxial $IrSi_3$ growth mode at ~700 °C, found that the $IrSi_3$ epitaxy on Si(111) was dominated by a Mode B* orientation which had not previously been reported in the literature, as well as showed that the epitaxial growth of $IrSi_3$ on Si(111) was superior to that on Si(100). Measurements made at Rome Laboratory found that the Schottky barrier height of the $IrSi_3$ film on Si(111) was considerably larger than that on Si(100) substrates. We also finished our detailed growth and structure studies of five different types of iridium silicide films: co-deposited and reacted IrSi, co-deposited Ir_3Si_4, reacted $IrSi_x$, co-deposited $IrSi_3$, and pure Ir reacted on hot Si substrates. This allowed us to form a previously-unreported silicide, Ir_3Si_4, and identify six epitaxial growth modes of Ir_3Si_4 crystallites with the Si(100) surface. At the end of this contract the student working on this research, Davis Lange, received his Ph.D. degree in Optical Sciences, and took a job working on infrared detectors at Loral Fairchild's laboratory in New York.

GROWTH AND CHARACTERIZATION RESULTS FOR IRIDIUM SILICIDES

I. Introduction

Achieving high quality silicon-silicide interfaces in iridium silicide is on of the principle problems of this materials system. For this reason, we conducted detailed characterization of the samples produced as part of this research program. For example, information is required with better than a few Ångstroms resolution about the structure, interdiffusion, sharpness, chemical purity, and electronic properties of the interfaces. The *in situ* analysis instruments that are part of our two MBE systems (XPS, ion mill Auger, and ISS) had the necessary sensitivity and depth resolution for the composition analysis of the iridium silicide surfaces we prepared as part of this research projects. Also, RHEED allowed us to determine the crystalline symmetry of the surface of the silicide films, and quantitative LEED studies allowed quantitative determination of the surface and interface atomic positions. Cross-sectional TEM studies provided both the required near-atomic-resolution images as well as microdiffraction patterns from the interfacial regions, and Atomic Force Microscopy (AFM) determined the surface roughness with equivalent resolution.

II. Summary of Our Most Significant Results

We used Molecular Beam Epitaxy to study the growth of $IrSi_3$ films on Si(100) and Si(111) substrates. $IrSi_3$ was produced by co-depositing Ir and Si in a 1:3 atomic stoichiometry to aid in the formation of pure, single phase films. We chose $IrSi_3$ as our first material to study due to the high barrier height reported when grown on n-type Si(100). For this work, we grew and studied films of both 450 Å and 100 Å thickness. We used Seemann-Bohlin x-ray diffraction, which is sensitive to the randomly oriented grains in these films, to determine the phase of our polycrystalline samples. During this work, we found we could form pure $IrSi_3$ films on both Si(100) and Si(111) substrates at temperatures as low as 450 °C; which is almost 200 °C lower than previously reported. Transmission Electron Diffraction (TED) allowed us to verify the purity of these polycrystalline $IrSi_3$ films.

Unfortunately, Bragg-Brentano x-ray diffraction is sensitive only to planes which are parallel to the substrate surface. However, we were able to use this technique to determine possible epitaxial growth modes as a function of deposition temperature for our co-deposited $IrSi_3$ films on Si(100) and Si(111). We developed a normalization procedure which accounts for the structure and Lorentz polarization factors, and used it to deduce the relative quantities of the

various seven possible epitaxial orientations (termed "modes" in the rest of this discussion) on Si(111) and three on Si(100) identified from peaks in our spectra. We determined that the epitaxial growth of $IrSi_3$ on Si(111) is superior to that on Si(100), and that the $IrSi_3$ epitaxy on Si(111) was dominated by the Mode B* orientation which had not previously been reported in the literature. We verified the existence of this epitaxial orientation using LEED, RHEED, and TED. Above 780 °C we found another epitaxial orientation to exist in large quantities, which we designate as Mode A. In addition, we found epitaxial growth for $IrSi_3$ on Si(100) in samples deposited between 630 °C and 780 °C, where the orientation of the $IrSi_3[2113]\|Si[100]$ and $IrSi_3[0110]\|Si[110]$ defines a previously unreported epitaxial growth mode for $IrSi_3$ crystallites on Si(100). This "Mode K" crystallite orientation does not produce a matching plane with the Si(100) surface since the (hkil) index of this plane is irrational, explaining why no evidence of this epitaxial growth was identified by our Bragg-Brentano analysis.

We fabricated a shadow mask from Mo with a number of 1/16" diameter diode areas for characterization of these films as infrared photodetectors. Measurements made at Rome Laboratory, given in our previous Technical Report of 10 January 1995, showed that the Schottky barrier height of the $IrSi_3$ film on Si(111) was considerably larger than that on Si(100) substrates. Unfortunately, the barrier heights found for $IrSi_3$ on Si(111) of around 0.33 eV and on Si(100) of 0.175 eV would not allow imaging in the Long Wavelength InfraRed (LWIR) of 8–12 μm. Detection of photons in this region requires barrier heights lower than 0.155 eV. However, a device with a barrier height of 0.175 eV could be useful in the Medium Wavelength InfraRed (MWIR) of 3–5.4 μm where PtSi detectors now are generally used. This would be of great value if the performance in the 4.5–5.4 μm window could be made greater than a PtSi detector. Studies involving tuning the thickness and deposition temperature of this material to provide maximum emission efficiency would be required to explore this possibility.

We also studied the growth and structure of five different types of iridium silicide films: codeposited and reacted IrSi, codeposited Ir_3Si_4, reacted $IrSi_x$, codeposited $IrSi_3$, and pure Ir reacted on hot Si substrates. We found Seemann-Bohlin x-ray spectra for polycrystalline formation of these iridium silicide films to be unique, and TED allowed us to verify that these films are different in structure. Atomic Force Microscopy (AFM) showed the surface of the Ir_3Si_4 film to be much smoother than the similarly processed IrSi film. The AFM measurements of the Ir_3Si_4 film showed a 3 Å RMS roughness and of the IrSi film a 12 Å RMS roughness. In both cases, the samples contained approximately 50 Å of iridium. We confirmed the smooth surface of the

Ir_3Si_4 film using Transmission Electron Microscopy (TEM) on a cross section of the above film.

Ir_3Si_4 co-depositions on Si(100) at temperatures between 450 °C and 550 °C form localized epitaxial films, although the TED pattern shows the presence of six epitaxial growth modes of Ir_3Si_4 crystallites with the Si(100) surface, where the primed indexing refers to a 90 degree rotation on the Si(100) surface of the same crystallite orientation. The Ir_3Si_4 films formed by annealed reactions are polycrystalline on both Si(100) and Si(111) substrates. The d-spacings measured from the diffraction patterns of these polycrystalline films are consistent with the Ir_3Si_4 crystal structure.

Measurement made at Hanscom AFB of an Ir_3Si_4 film deposited on a hot silicon substrate at 450 °C revealed fairly good infrared detector characteristics as shown in Figure 12. The C_1 value of > 9 %/eV and cutoff wavelength in the LWIR of 8.9 μm shows promise for LWIR detectors to be developed from this film type. Measurements also were made at Hanscom AFB on our annealed Ir_3Si_4 films. The data from these films showed slightly lower barrier heights out to ~10 μm. However, they displayed lower C_1 values than that of the hot- deposited film.

Our work also showed that silicides formed when pure Ir films were deposited on hot Si substrates at ~500 °C. From peaks observed in x-ray diffraction, we were able to confirm Ir_3Si_4 growth in these reactions, and TED confirmed that the Ir_3Si_4 films formed by Ir depositions at 500 °C contain epitaxial Ir_3Si_4 growth. The electron diffraction patterns clearly showed the formation of the Ir_3Si_4 A, B, and C localized epitaxial growth modes on Si(100) as described above.

We measured the electrical properties of our iridium silicide films using the 1/16" diodes discussed above. By measuring the current at a fixed reverse bias voltage in the range 0.1–1.0 volts, and plotting $\ln(I/T^2)$ vs. $1/k_BT$ we could obtain the thermal barrier height of the devices. Here I is the current measured, T is the temperature of the device, and k_B is Boltzman's constant. We measured an extremely low barrier height of 0.1 eV for an Ir_3Si_4 sample which was deposited at ambient and subsequently annealed to 500°C.

PUBLICATIONS

1. **Growth of MBE-Codeposited $IrSi_3$ on Si(111) and Si(100).** Gary A. Gibson, Davis A. Lange and Charles M. Falco, Proc. of the Materials Research Society 299 (1993).

2. **Metallic Multilayers and Superlattices.** Charles M. Falco, chapter in Encyclopedia of Applied Physics, edited by George L. Trigg (VCH Publishers, New York, 1993).

3. **MBE-Codeposited Iridium Silicide Films on Si(100) and Si(111).** Davis A. Lange, Gary

A. Gibson and Charles M. Falco,. of the SPIE. 2021, 67 (1993).

4. **Growth and Structure of $IrSi_3$ on Si(111).** Davis A. Lange, Gary A. Gibson and Charles M. Falco, J. Appl. Phys. 75, 2917 (1994).

5. **Electron Beam Evaporator Based MBE.** Erich Kasper and Charles M. Falco, chapter 3 in Advanced Silicon and Semiconducting Silicon-Alloy Based Materials & Devices, edited by J. F. A. Nijs (IOP, London, 1994).

6. **Quantitative Analysis of Bragg-Brentano Data for Highly-Oriented Thin-Film Samples.** Gary A. Gibson, D. A. Lange and Charles M. Falco, J. Appl. Phys - SUBMITTED.

7. **MBE-Deposited Iridium Silicides for Focal Plane Array Applications.** Davis Alan Lange, Ph.D. Dissertation, University Microfilms, 1996.

STUDENTS AND POSTDOCS WORKING ON THIS CONTRACT

Dr. Gary Gibson worked on this contract for its first two years. He left in the Spring of 1993 to take a full-time position as member of the technical staff at Hewlett-Packard Research Laboratories in Palo Alto.

Davis Lange worked on this contract from the start, as part of his Ph.D. dissertation research in Optical Sciences. He defended his Ph.D. on 16 November 1995, and took a job working on infrared detectors at Loral Fairchild Systems.